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भाग 8 सीलीकॉन अंश निर्धारण

Indian Standard

NICKEL ALLOYS — FLAME ATOMIC ABSORPTION SPECTROMETRIC ANALYSIS — METHOD

PART 8 DETERMINATION OF SILICON CONTENT

ICS 77.120.40

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 2000 **Price Group 2**

Methods of Chemical/Instrumental Analysis of Non-ferrous Metals Sectional Committee, MTD 28

NATIONAL FOREWORD

This Indian Standard (Part 8) which is identical with ISO 7530-8:1992 'Nickel alloys — Flame atomic absorption spectrometric analysis — Part 8 : Determination of silicon content' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Methods of Chemical/Instrumental Analysis of Non-ferrous Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

The text of the above mentioned ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 5725 : 1986	IS 5420 (Part 2) : 1973 Guide on precision of test methods: Part 2 Inter-laboratory testing	Not Equivalent
ISO 7530-1 : 1990	IS 14644 (Part 1) : 1998 Nickel alloys — Flame atomic absorption spectrometric analysis: Part 1 General requirements and sample dissolution	Identical

This standard has been issued in nine parts. Other parts of this standard are :

Part 1	General requirements and sample dissolution
Part 2	Determination of cobalt content
Part 3	Determination of chromium content
Part 4	Determination of copper content
Part 5	Determination of iron content
Part 6	Determination of manganese content
Part 7	Determination of aluminium content
Part 9	Determination of vanadium content

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

NICKEL ALLOYS — FLAME ATOMIC ABSORPTION SPECTROMETRIC ANALYSIS — METHOD

PART 8 DETERMINATION OF SILICON CONTENT

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of silicon in the range of 0,2 % (*m*/*m*) to 1 % (*m*/*m*) in nickel alloys. Typical compositions of nickel alloys are given in annex B of ISO 7530-1:1990.

The general requirements concerning the apparatus, sampling, dissolution of the test sample, atomic absorption measurements, calculations and test report are given in ISO 7530-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7530. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 7530 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 7530-1:1990, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: General requirements and sample dissolution.*

3 Principle

Dissolution of a test portion in acid and aspiration of the test solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer.

Measurement of the absorbance of the resonance line energy from the spectrum of silicon and comparison with that of calibration solutions at a wavelength of 251,6 nm.

4 Reagents

In addition to the reagents listed in ISO 7530-1, the following special reagents are required.

4.1 Hydrofluoric acid, $\rho_{20} = 1{,}15$ g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water and seek medical advice.

4.2 Hydrofluoric acid, $\rho_{20} = 1{,}15$ g/ml, diluted 1 + 9.

4.3 Lithium chloride (LiCl), solution.

Transfer 25 g of lithium chloride to a 250 ml beaker and dissolve in 150 ml of warm water. Cool, transfer to a 200 ml one-mark volumetric flask and make up to the mark with water. Store in a plastics bottle.

4.4 Silicon, standard reference solution (1,000 g/l).

Weigh, to the nearest 0,001 g, 1,000 g of elemental silicon powder of 99,9 % (*m*/*m*) minimum purity and transfer to a 250 ml polytetrafluoroethylene beaker. Add 20 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml) and wash the beaker walls with water. Add hydrofluoric acid (4.1) drop by drop to initiate and sustain the reaction (approximately 10 ml of hydrofluoric acid are required). After most of the silicon has dissolved, add

10 ml more hydrofluoric acid, cover the beaker and keep at less than 50 °C until dissolution is complete. Transfer to a 1 000 ml plastics one-mark volumetric flask, add 20 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and make up to the mark with water. Store in a polyethylene bottle.

4.5 Silicon, standard solution (100 mg/l).

Transfer, using a plastics pipette, 50,0 ml of the silicon standard reference solution (4.4) into a 500 ml plastics one-mark volumetric flask. Add 5 ml of the dilute hydrofluoric acid (4.2) and 10 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Dilute to the mark with water, mix and store in a polyethylene bottle.

5 Apparatus

IMPORTANT — Plastics beakers and volumetric ware shall be used throughout the procedure.

In addition to the apparatus specified in clause 5 of ISO 7530-1:1990, the following plastics laboratory items are required.

5.1 Plastics beakers, of capacity 250 ml, preferably of polytetrafluoroethylene.

5.2 Acrylic body burette, 50 ml graduated in 0,1 ml divisions.

5.3 Polypropylene pipettes, 10 ml, 25 ml and 50 ml.

5.4 Polypropylene or polymethylpentene one-mark volumetric flasks, of capacity 100 ml, 500 ml or 1 000 ml.

6 Sampling and sample preparation

Refer to clause 6 of ISO 7530-1:1990.

7 Procedure

7.1 Preparation of test solution

7.1.1 Dissolution of a test portion in acid

Weigh, to the nearest 0,001 g, 1,00 g of the test sample and transfer it to a clean plastics beaker (5.1). Add 20 ml of a mixture of one part of nitric acid (ρ_{20} = 1,41 g/ml) and three parts of hydrochloric acid (ρ_{20} = 1,18 g/ml). Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, add hydrochloric acid (ρ_{20} = 1,18 g/ml) in 1 ml increments and continue heating to dissolve the sample.

7.1.2 Preparation of the final test solution

Cool the solution and wash the cover and beaker walls with a minimum of water. Add 5 ml of the dilute hydrofluoric acid (4.2) and allow to stand for 1 h, swirling intermittently.

7.1.3 Primary dilutions

7.1.3.1 Initial dilution for 0,2 % (*m*/*m*) to 0,25 % (*m*/*m*) silicon

Transfer the test solution from 7.1.2 to a 100 ml plastics one-mark volumetric flask, add 2 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and dilute to approximately 80 ml with water. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water.

7.1.3.2 Initial dilution for 0,5 % (*m*/*m*) to 1,0 % (*m*/*m*) silicon

Transfer the test solution from 7.1.2 to a 100 ml plastics one-mark volumetric flask and make up to the mark with water.

7.1.4 Secondary dilution for 0,5 % (*m*/*m*) to 1,0 % (*m*/*m*) silicon

Pipette 50,0 ml of the solution from 7.1.3.2 into a 100 ml plastics one-mark volumetric flask. Add 2 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 2,5 ml of the dilute hydrofluoric acid (4.2). Dilute to approximately 80 ml with water and mix. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water.

7.2 Reagent blank solution

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents.

7.3 Silicon calibration solutions

Using the plastics burette (5.2), transfer to each of six 100 ml plastics one-mark volumetric flasks, 0 ml, 10,0 ml, 20,0 ml, 30,0 ml, 40,0 ml and 50,0 ml of the silicon standard solution (4.5). Add 2 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 5 ml of the dilute hydrofluoric acid (4.2). Dilute to approximately 80 ml with water and mix. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water. These calibration solutions correspond to 0 mg/l 10 mg/l, 20 mg/l, 30 mg/l, 40 mg/l and 50 mg/l of silicon.

7.4 Calibration and determination

7.4.1 Atomic absorption measurements

Proceed as directed in 7.4.1 of ISO 7530-1:1990, using a wavelength of 251,6 nm and a nitrous oxide-acetylene flame.

NOTE 1 To eliminate silica memory effects, the burner system must be preconditioned before analysis by aspirating a dilute solution of hydrofluoric acid [10 ml of dilute hydrofluoric acid (4.2) and 90 ml of water]. With the flame burning, aspirate this dilute acid solution until the original baseline signal is restored, i.e. when the silica deposit on the burner top has been volatilized. Then proceed with the aspiration of distilled water as directed.

7.4.2 Preparation of calibration curves

Proceed as directed in 7.4.2 of ISO 7530-1:1990.

7.5 Number of determinations

Carry out the determination at least in duplicate.

8 Expression of results

8.1 Calculation

Proceed as directed in 8.1 of ISO 7530-1:1990.

8.2 Precision

8.2.1 Laboratory tests

Six laboratories in four countries participated in the testing of this procedure using six samples of nominal composition given in table 1.

8.2.2 Statistical analysis

8.2.2.1 Results were treated according to ISO 5725 as described in 8.2.2 of ISO 7530-1:1990. The results of this analysis are given in table 2.

8.2.2.2 No outliers were identified by statistical tests.

9 Test report

Refer to clause 9 of ISO 7530-1:1990.

Table 1 — Nominal composition of test samples [% (*m*/*m*)]

Sample	Al	Co	Cr	Cu	Fe	Mn	Ni	Si	Ti
825	0,2	0,07	21	1,6	30	0,7	Bal	0,4	1,1
902	0,4	0,05	5	0,04	48	0,4	Bal	0,35	2,5
3920	0,15	2	19	0,1	3	0,3	Bal	0,6	2,3
3927	0,1	1	20	0,05	44	0,4	Bal	0,8	0,6
7013	1,5	17	20	0,2	0,2	0,05	Bal	0,7	2,4
7049	1	0,01	15	0,15	7	0,8	Bal	0,3	2,3

Table 2 — Results of statistical analysis

Sample reference	Mean % (*m*/*m*)	Within-laboratory standard deviation	Between-laboratory standard deviation	Repeatability	Reproducibility
825	0,403	0,006 6	0,023 6	0,018 5	0,069 4
902	0,344	0,003 9	0,014 9	0,011 0	0,043 5
3920	0,614	0,016 6	0,017 5	0,047 0	0,068 2
3927	0,816	0,019 5	0,040 3	0,055 1	0,127
7013	0,721	0,017 4	0,021 3	0,049 1	0,077 8
7049	0,336	0,006 6	0,014 4	0,018 7	0,044 8

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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